CORESTA

SMOKE STUDY GROUP

Montreux, September 23-24, 1974

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COOPERATION CENTRE FOR SCIENTIFIC RESEARCH RELATIVE TO TOBACCO

SCIENTIFIC COMMISSION

Smoke Study Group

MEETING IN MONTREUX - SWITZERLAND

September 23-24, 1974

A G E N D A

1. Report of the Pesticide Sub-Group (Mr. Gaisch).
2. Report of the Task Force on vapour phase components (M. Joigny).
3. Report of the Task Force on metals in smoke (Mr. Osdene).
4. Election of President, Secretary and Candidates for Scientific Commission.
5. Report of the Cigar Sub-Group (Mr. Vander Straeten).
6. Standard Method - Moisture in tobacco (Messrs. Carugno / Lipp).
7. Report on questionnaires regarding butt length standards (Mr. Williamson).
8. ISO / CORESTA.
9. Future activities.
10. Joint meeting of Smoke and Technology Groups.
11. Place and date of next meeting.
12. Presentation of papers.

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LIST OF PARTICIPANTS OF THE CORESTA SMOKE STUDY GROUP

SEPTEMBER 1974

Country	Member Body	Name
AUSTRIA	Austria Tabakwerke A.G.	H. Klus
	" " "	H. Kuhn
AUSTRALIA	Rothmans of Pall Mall (Australia) Limited	W.P. Ryan
BELGIUM	C.E.E. Direction Générale de l'Agriculture (Division Tabac)	G. Rossetto
BULGARIA	Tobacco Research Centre, Plovdiv	Dr. N. Ivanov
CANADA	Rothmans of Pall Mall	N. Cohen
CZECHOSLOVAKIA	Research Institute of the Tobacco Industry	E. Nescak
EGYPT	Eastern Tobacco Company	A. Mostafa
ENGLAND	Cigarette Components Ltd.	R.T. Stephenson
	" " "	J.T. Williamson
	" " "	Mrs. M. Saran
	B. A. T., UK	C.I. Ayres
	Carreras, Rothmans Limited, Research Division	B.E. Frost
	Imperial Tobacco Limited	D.T. Westcott
FINLAND	P. C. Rettig and Company	K. Nummelin
FRANCE	JOB	L. Fauque
	S.E.I.T.A.	A. Testa
	"	C. Joigny

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Country	Member Body	Name
GERMANY	Landesanstalt für Tabakbau u. Tabakforschung	F.A. Schmidt
	Hauni Werke, Körber und Co. K.G.	A. Helms
	A.N. Hertz	W. Weitz
	Dr. Rhodiaceta A.G.	D. Tueberg
	Martin Brinkmann A.G.	M. Gehring
	B. A. T. Cigarettenfabriken GmbH	W. Schulz
	Reemtsma, Wissenschaftliche Abt.	E.U. Kaussmann
	Martin Brinkmann A.G.	H. Elmenhorst
HOLLAND	Philip Morris Holland B.V.	J.W. Osborne
	D. E. J. International Research Company B.V.	D. Schurer
	Turmac Tobacco Company B.V.	J. van der Heide
HUNGARY	Institut d. Rech. du Tabac	Dr. L. Nemethi
INDONESIA	Deli Tobacco Research Station	A.T. Tojib
IRAQ	Iraqi State Tobacco Company	J. Bashir
	" " " "	I. Kamal
ITALY	Amministrazione Monopoli di Stato	N. Carugno
	Ass. Produttori Tabacchi Italiani	I. Caprio
JAPAN	Japan Tobacco and Salt Public Corporation	N. Takemoto
LIBYA	General Tobacco Company	M. El Mabruk
LUXEMBOURG	Manufacture de Tabacs Heintz van Landewyck Sarl	C. Braun
SOUTH AFRICA	Rupert International (Pty) Ltd	J.D. Malan
SWEDEN	Swedish Tobacco Company	L.O. Hjern
	" " "	C.R. Enzell

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Country	Member Body	Name
SWITZERLAND	Baumgartner Papiers S.A.	J. Bonnet
	Fabriques de Tabac Réunies S.A.	C. Jeanneret
	" " " " "	H. Gaisch
	F. J. Burrus et Cie	R. Koch
THAILAND	Thailand Tobacco Monopoly, Research Department	S. Suebsaeng
U. S. A.	Lorillard, A division of Loews Theatres Inc.	F. Schultz
	" " "	A.W. Spears
	U. S. Tobacco	W.B. Bennett
	R. J. Reynolds Tobacco Company	M. Senkus
	Tennessee Eastman Company	G. Touey
	Liggett and Myers	V. Norman
	Philip Morris U.S.A.	T.S. Osdene
	USDA Agriculture Research Service Tobacco Storage Research Unit	D.P. Childs
YUGOSLAVIA	Tobacco Institute	T. Grabiloski

Only the names of representatives of member companies which were present during the business meeting and at the election procedure are given in this list.

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CORESTA SMOKE STUDY GROUP - REPORT ON WORKING SESSIONS

Mr. Elmenhorst welcomed the members and opened the meeting with a brief review of the programme.

1. Report of the Pesticide Sub-Group

The Sub-Group held meetings in December 1973 and in May 1974, at which much useful work was done. Mr. Gaisch particularly commented on the great assistance received from Mrs. Cora Ayeres since she took up duties as secretary to the Group.

The Group has done much to establish the limitations of what is practically possible and to determine the source of interference in the methods. As many as sixteen laboratories have taken part in the last joint experiment and methods are available for the determination of organochlorine, dithiocarbamate and some organophosphorous pesticides. The CORESTA recommended Methods N. 1 and 2 on dithiocarbamates and chlorinated hydrocarbon residues have been published in the CORESTA Bulletin.

A more universal method which has been developed by Nesemann for analysing a wide range of organophosphorous compounds and carbamate, including both genuine pesticides, metabolites and other break-down products, is being critically tested by Sub-Group members.

Mr. Gaisch added that although many basic facts are documented in the reports of the Sub-Group, the real know-how is much wider and more comprehensive and resides in the experience of the Sub-Group members. Consequently, if there are pesticide problems to tackle, it is the Sub-Group which is most qualified to do so.

Next meeting of the Sub-Group will be in Neuchatel, February 1975.

2. Report of the Task Force on vapour phase components

M. Joigny submitted an extensive report , in which he reviewed definitions, problems encountered and methods of determination. The principle methods covered were for CO, oxides of nitrogen, acrolein, HCN, sulphur compounds, together with a general method using high resolution gas chromatography for a wide range of other compounds.

A full reprint of M. Joigny's report is given as Appendix I.

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3. Report of the Task Force on metals in smoke

Mr. Osdene reported that since the last meeting he had received eight further references. To date, 257 references are listed, but most concern trace metals in tobacco, rather than in smoke. He especially referred to a paper by Martell describing the retention of Lead 210 on the trichomes of tobacco leaves while growing in the field, as this may have some significance. Mr. Osdene again requested that members continue to send him references as they appear.

4. Election of President, Secretary and Candidates for Scientific Commission

Mr. Elmenhorst chaired the meeting which was attended by 55 members from 25 countries, as indicated in the attached list. Nominations had been requested in advance and were accepted up to just before voting commenced. The voting was as follows

	Candidates	No. of Votes
FOR PRESIDENT	Elmenhorst	36
	Häusermann	7

Mr. Elmenhorst was duly elected President.

FOR SECRETARY (the current Secretary being unable to continue)	Osdene	11
	Westcott	7
	Morie	3
	Hjern	8
	Viart	15

Mr. Viart was duly elected Secretary.

PROPOSALS FOR CANDIDATES FOR THE SCIENTIFIC COMMISSION	Dietrich	4
	Kuhn	16
	Viart	11
	Artho	30
	Gaisch	5
	Spears	10
	Schultz	8
	Westcott	3
	Ayres	8
	Häusermann	9
	Senkus	16
	Morie	12

Messrs. Artho, Kuhn and Senkus were proposed as Candidates for the Scientific Commission, along with the President - Mr. Elmenhorst.

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5. Report of the Cigar Sub-Group

Mr. Vander Straeten reported on the work of the Cigar Study Group, as a consequence of which certain propositions had been put forward as the basis of a standard method for cigar testing. Further consideration was, however, to be given to certain points regarding acceptability by Technology Group members.

These points were discussed in a short meeting of members of the Cigar and Technology Groups on September 24, and agreement had been reached on the following:

1. Cigars are smoked differently from cigarettes.

2. The parameters proposed by the Cigar Smoke Sub-Group for laboratory smoking of cigars between 1.5gm. and about 5.5gm in weight, are acceptable as a basis for studying the smoke of cigars.

3. Further collaborative studies should be conducted to define the methods of determination of smoke composition. The Cigar Smoke Sub-Group would welcome comparative information on cigar smoking from other laboratories, particularly on methods used for analysis. These informations could be sent to Mr. Vander Straeten or Mr. Edge.

6. Standard Method - Moisture in tobacco

Between November 1973 and February 1974, a collaborative study was made between seventeen laboratories to examine the revised distillation method using cyclohexane in place of benzene. One laboratory prepared all samples of each of three types of tobacco, Virginia, Blend and Stems, with different moisture content and six determinations were carried out on each type. Fourteen of the laboratories agreed within a range of 0.33% moisture and it is proposed that the method be considered for adoption as a CORESTA Standard Method.

A short report by Mr. Lipp regarding this study is attached as Appendix II. A complete report will be published in the BEITRÄGE ZUR TABAKFORSCHUNG. Mr. Carugno suggested that a final draft of the method should be sent to all members who will send comments to the secretary. A final text will then be prepared for approval at the next meeting.

7. Report on questionnaires regarding butt length standards

A proposition by Mr. Häusermann made at Nice that we should consider changing the CORESTA standard butt length to a value nearer the average used most widely in a number of countries where Government testing was in existence, was dealt with by sending questionnaires to all Smoke Study Members and asking them:

1. to report the standard butt lengths used in their country, and

2. whether they felt a change from the current CORESTA Standard was desirable.

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Thirty two replies were received for item 1. above, from which it appeared that in the majority of cases a non-filter butt length of 23mm. is used and this is also employed as a minimum butt length for filters. For longer butts, the butt length of a filter cigarette is determined in the majority of cases as either filter length plus 8mm., or tipping length plus 3mm.

Thirty five replies were received to item 2. above, from which it was clear that the majority wished to make no change in the present CORESTA standards on butt lengths.

(Secretary's letters of 5th December 1973 and 8th March 1974 refer.)

8. ISO / CORESTA

The Pesticide Sub-Group has developed a method for dithiocarbamate residues and another method for chlorinated hydrocarbon residues on tobacco. These are printed in the CORESTA Bulletin as CORESTA Recommended Method N. 1 and 2. The two methods will be offered to ISO for consideration as standard methods at the next meeting of ISO.

9. Future activities

Mr. Elmenhorst reviewed the points outstanding from their Rome meeting referring to the work of the Pesticide, Gas Phase and Materials Working Parties and asked for any other suggestions for future group work. No immediate suggestions were forthcoming, but Mr. Elmenhorst suggested that further ideas should be sent to the Secretary (M. Viart) at any time, in writing.

10. Joint meeting of Smoke and Technology Groups

Three papers were presented, numbers 56, 57 and 58 - see attached list of titles. Following reports of the Smoke Study Group's activities and the Technology Group's activities by the respective secretaries, Mr. Elmenhorst indicated that a final draft for the Cigar Study Group's proposals for a standard smoking parameters for cigars had been resolved after a meeting with Mr. Vander Straeten and the members of the Technology Group who had objected to the original draft.

He asked if there any new suggestions or objections to this, and as there were none he advised that it would be put to the Scientific Commission as a recommended standard.

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11. Place and date of next meeting

It was announced that an invitation had been received from Scantoco for the 1975 meeting, to be held in Copenhagen. The meeting would be in September and the precise dates would be announced later. *)

12. Presentation of papers

Seventeen papers were presented to the Group. See attached list of paper titles.

Abstracts of all the papers presented are given in the CORESTA Information Bulletin 1974 special issue.

*) September 5 and 6 Smoke Study Group meeting
September 2 and 3 Technology Group meeting

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PAPERS PRESENTED AT THE CORESTA SMOKE STUDY GROUP MEETING

SEPTEMBER 1974

No.	Title and Author
28	The effect of climatic conditions on smoke. DYMOND H. F. D.
29	Parameters affecting the selective filtration of certain tobacco smoke components. MORIE G. P., SLOAN C. H., BAGGETT M. S.
30	Separation of nicotine from weak aqueous solutions by ion exchange membrane electrodialysis. TAKEMOTO N., SHIMIZU Y., TAMAKI E.
31	The determination of nicotine in tobacco samples by gas chromatography. VAN DER HEIDE J.
32	Alkaloid determination in tobacco. WAHL R.
33	Carbon monoxide determination in the gas phase of tobacco smoke. GRUNWALD G.
34	Isolation and identification of volatile components of flue-cured tobacco. LLOYD R. A., MILLER C. W., ROBERTS D. L., GILES J. A., DICKERSON J. P., NELSON N. H., RIX C. E., AYERS P. H.
35	Nonvolatile nitrosamines in tobacco smoke. An experimental approach. KLUS H., KUHN H.
36	Nonvolatile N-nitrosamines in tobacco and tobacco smoke. HOFFMANN D., HECHT S.S., ORNAF R.M.
38	Effects of cigarette length on the chemical composition of tobacco smoke. GRABILOSKI T., PERUSESKA C.
39	Treatment of tobacco with enzymes: its effects upon certain leaf components and upon cigarette smoke. CARUGNO N.
40	Studies in view of the analytical determination of the degree of "sharpness" of tobacco. EGRI L.
41	Effects of the incorporation of adsorbing agents into reconstituted tobacco upon the smoke composition. BERINGER M., KOCH R.
(No number)	A smoking machine for the analysis of vapour phase compounds. FROST B., CUMMINGS E. M.

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PAPERS PRESENTED AT THE JOINT MEETING OF THE SMOKE AND TECHNOLOGY GROUPS

No.	Title and Author
56	Some relations between surface properties and the filtration of total tobacco smoke. JEANNERET C., NYFFELER U., MAYOR C., SAUSER M. F.
57	The design and development of a light scattering photometer for the size analysis of cigarette smoke. HILL C.A., ADAMS P. I.
58	Sensory analysis of cigars. HALTER H. M., ITO T. I.

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Appendix I

REPORT OF THE TASK FORCE

ON THE GAS PHASE OF THE CIGARETTE SMOKE

CORESTA Conference - MONTREUX - September 23-27, 1974

C. Joigny

I - INTRODUCTION : THE GAS PHASE OF CIGARETTE SMOKE

I.1 - Definition - Preparation

I.2 - Problems of Aging - Conservation

I.3 - Drawbacks and Advantages of Determinations on the Gas Phase

I.4 - Methods of Determination Used

II - PRINCIPAL COMPOUNDS NOW DETERMINED IN THE GAS PHASE OF CIGARETTE SMOKE

II.1 - Carbon Monoxide CO

II.2 - Nitrogen Oxides NO - NO_2

II.3 - Acrolein

II.4 - Hydrogen cyanide HCN

II.5 - Sulphur compounds

II.6 - Determination of Various Components of the Gas Phase by High Resolution Gas Phase Chromatography (GC)

III - CONCLUSION

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I - INTRODUCTION : THE GAS PHASE OF CIGARETTE SMOKE

I.1 - Definition - Preparation:

As cigarette smoke is an aerosol it is theoretically possible to distinguish a gas phase and a particulate phase. In practice, the problem of the separation of the two phases is less simple: any screen capable of retaining particles larger than a certain diameter allows a high proportion of smaller particles to pass with the gases and vapours. Furthermore, as puffs succeed one another, the filter becomes partially blocked so that, during an aspiration, certain semi-volatile products dissolved in the condensate will pass through the filter, whereas (which is more troublesome in gas phase determinations) certain volatile products could be dissolved by the condensate. The great complexity of the equilibria involved means that the gas phase is defined empirically and rather arbitrarily as being "that which passes" through a glass fibre filter of well defined mesh size, known as a Cambridge filter, placed immediately behind the smoked cigarette. Tobacco moisture content, the conditions of temperature and moisture obtaining in the smoking room, the type of smoking machine and how it is adjusted should also, naturally, be carefully defined. This *a priori* definition of the smoke phase is to be the subject of an ISO standard, and what we refer to as the "gas phase" in the following article is the fraction of smoke separated according to this standard.

I.2 - Problems of Aging - Conservation:

Most of the compounds found in the gas phase which are of interest in determinations are reactive compounds (NO_x, acrolein, H_2S, HCN): it is therefore desirable to carry out determinations on the compounds themselves as quickly as possible (by direct injection in GC or by non-dispersive spectrometry) or to convert them very quickly into stable addition compounds. Only the oxides of carbon (CO - CO_2) require no precautions, except for the material of the container in which they are stored. Furthermore, the metal portions of the apparatus will catalyse the reactions of a given product more efficiently, causing it to disappear. The articles by GROB (2) and SCHÖNHERR *et al.* (4) deal with the problems of gas phase denaturation.

I.3 - Drawbacks and Advantages of Determinations on the Gas Phase

The drawbacks are principally methodological:

a) Is the mixture of gas and vapours known as the "gas phase" constant for a given type of cigarette? Provided that the precautions mentioned in § I.1 are taken, the answer is: yes, insofar as two cigarettes of the same type are identical.

b) Does the gas phase contain the total amount of the products or does a certain proportion remain in the particulate phase? This reproach is theoretically serious as, if the

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percentage of the component of the total smoke passing into the gas phase is not known, determination in the gas phase is of little interest. With respect to this problem, for example, reference may be made to the articles by WALTZ & HAUSERMANN (5) and WILLIAMSON & ALLMAN (6). This is why we have restricted our study to compounds which are sufficiently volatile for it to be assumed that large proportions of them exist in the gas phase, although definite indications do not always exist as to their distribution in the two phases.

The advantages are of a more practical nature:

- the relative simplicity of this mixture of volatile products, which does not contain resinous products (a great advantage for colorimetric analysis and gas chromatography, where there is no short term threat to the columns);

- owing to this "cleanliness", the possibility of analysing "native" products (with a time lag which may not be more than a few seconds for analysis of one puff, which reduces the risks of destroying the compound) and of using spectrometric or colorimetric methods without excessive absorption.

To sum up, although the gas phase is not smoke, in the case of important compounds, its analysis makes it possible to obtain:

- in certain cases, a value which can be taken as being identical to that of the whole smoke (CO, NO, H_2S);

- in other cases, an indicative value (but which has the virtue of existing) which enables:

. comparisons to be made between different treatments, manufacturing methods, mixtures of tobaccos, etc.

. exact information to be obtained on the concentration of a semi-volatile compound in the vapour phase, which can be important in the case of studies on absorbent filters, which affect compounds in the vapour state.

The qualitative composition of the gas phase has been dealt with first by WYNDER & HOFFMANN (7), then in greater detail by ELMENHORST & SCHULTZ (1). This review only deals with the methods for determining some of the major components of smoke present in the gas phase in their totality, or in large proportions, with some further information on chromatographic determinations of the gas phase taken as a whole. It is therefore, in a way, a thorough study of a limited number of compounds, as compared with SENKUS's review (3) which, as it deals with a very much larger number of compounds, had to be limited in the choice of references. For each compound studied, we shall try to give an idea of all the methods proposed and those which appear to be the most interesting at present.

I.4 - Methods of Determination Used:

The methods of determination used can be reduced to four types:

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- Spectrometry (dispersive or non-dispersive IR; UV) of the native compound.

  Advantages: rapidity and simplicity; secondary reactions reduced.

  Drawbacks: risks of interferences on the selected wavelengths; difficult to automate.

- Colorimetry on an addition compound.

  Advantages: automation possible (at least for measurement); can eliminate secondary reactions which would consume the compound to be determined.

  Drawback: risk of interferences which are sometimes uncontrollable.

- Gas chromatography - on conventional column
  - on capillary column

  with various detectors (thermal conductivity, flame ionization, flame photometry, thermionic, electrolytic conductivity, etc.).

  Advantages: "visualisation" of the mixture, therefore relative specificity, especially with specific detectors (flame photometry, thermionic or electrolytic conductivity); generally speaking, good reproducibility.

  Drawbacks: the difficulty of knowing whether a peak is not composite (except when a coupled mass spectrometer is available); the necessity of manually measuring peaks, or using an integrator (costly); special experience for capillary columns.

- Electrochemical methods (selective electrodes, potentiometry).

  Advantage: simplicity of measurement.

  Drawbacks: difficult to automate; may, furthermore, require a preliminary reaction which can take a certain amount of time.

II - PRINCIPAL COMPOUNDS NOW DETERMINED IN THE GAS PHASE OF CIGARETTE SMOKE

II.1 - Carbon Monoxide CO (B.P. = -191·5°C):

This is the easiest of the interesting products to determine in the gas phase: it is stable, hardly reacts under ambient conditions, and is a permanent gas. But certain precautions should be taken to avoid losses by diffusion when the gas phase is stored prior to determination [BRUNNEMANN & HOFFMANN (10), HORTON & GUERIN (14)]; the best containers appear to be bags made from "Saran" (polyvinylidene chloride) or "Metathene" (Polythene/polypropylene laminate) [AYRES (8)], but teflon and rubber are acceptable for analyses effected soon after smoking.

All the methods imply more or less frequent calibration. The ideal is to have bottles of gas (CO and/or CO_2 with a known

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content of nitrogen or helium). Failing this, CO may be produced by the action of sulphuric acid on formic acid [*cf.* KRUSZYNSKI & HENRIKSEN (17)].

Two types of methods compete for determination:

a) Chromatographic methods:

Taking into account the high concentration of CO in smoke (about 50,000 ppm., or 5%), the detector is a thermal conductivity detector (katharometer). KLIMISCH & MEISSNER (15) in a recent investigation nevertheless use flame ionisation detection, after reduction of CO to methane CH_4 on a nickel catalyst. The increased sensitivity thus obtained does not appear useful for determinations on "native" smoke (even puff by puff), but can become so for determinations on atmospheric samples, or for physiological studies on CO.

Conventional columns, 1/4 or 1/8 inch in diameter, are used. Analysis can take from 1 minute to 1 hour, depending on the temperature of analysis, length of the column, and the type of adsorbent (solid) used as packing, which may be:

- silica gel:

As CO is not separated from atmospheric gases, as opposed to CO_2, a small amount of iodic acid anhydride, which oxidises CO to CO_2, is placed in the middle of the column, which permits the permanent gases, CO and CO_2, to be finally separated. The fact that the analysis only lasts for 2 minutes, and the strength of the substrate, are two very positive elements of this method [WATANABE & KOBASHI (21); *cf.* also BRUNNEMANN & HOFFMANN (10); DONZEL (12)]. The I_2O_5 and Ag cartridge should probably be replaced every 200 to 300 determinations.

- carbon molecular sieves:

Described by MORIE (18): duration of analysis, 3 minutes for CO, 8 minutes for CO and CO_2, with temperature programmed at 30° to 100°C; then by KLIMISH (16): duration of analysis, less than 1 minute, which makes it possible to determine the CO in each puff of the same cigarette.

It should be noted that it is only necessary to carry out such rapid analyses for fundamental studies on cigarette combustion: in general, yield per cigarette is studied; in this case, the analysis should be quicker than smoking (about 10 minutes) or a fraction of the smoking time (1/2 or 1/3) if 2 or 3 replications of the measurement are required.

The advantage of carbon molecular sieves over zeolite molecular sieves is that they are not sensitive to moisture (and therefore do not necessitate desiccation of the gas phase). As the column is not very fragile, many analyses can be effected (about 1,000) without regeneration [KLIMISCH (16)].

- zeolite molecular sieves:

. type 5A: *cf.* KRUSZYNSKI & HENRIKSEN (17), HORTON & GUERIN (14); REIF & KUHN (20) (duration of analysis < 2 minutes for CO alone; about 30 minutes for O_2, CO and CO_2 with temperature programming to 340°C;

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. type 13 X: *cf.* KLIMISCH & MEISSNER (15), who determine CH_4 and CO in the form of CH_4 (after hydrogenation) in less than 2 minutes; AYRES (8) uses two columns in parallel, the zeolite column being used for the separation of CO from the permanent gases after absorption of CO_2 on soda lime (duration about 15 minutes for permanent gases, less for CO alone).

The sensitivity of zeolites to water results in most authors vigorously drying the gas phase (with $Mg(ClO_4)_2$ for example), and the high CO_2 retention level necessitates very strong heating [*cf.* REIF & KUHN (20)].

- cation exchange resin:

A cation exchange resin (Amberlite) in the Ni form, reduced with hydrogen: *cf.* HORTON & GUERIN (14). Analysis takes less than 4 minutes. The manufacture of adsorbent may pose problems (notably, reproducibility of Rf from one batch to another).

It should be noted that a method using porous resin (Porapak Q), with temperature programming from -70°C [MORIE & SLOAN (19)], has been given up by its authors in favour of a method on a carbon sieve [MORIE (18)], as the apparatus is cheaper and results are more easily reproducible.

b) Non-dispersive IR spectrometry method:

Although a non-dispersive IR apparatus costs about the same as a chromatograph a few laboratories now use them for determining CO, probably because of its specificity [which necessitates two analysers if CO and CO_2 are both to be determined; *cf.* WILLIAMS & BELK (22)].

The method is simple and analysis very rapid (it enables the evolution of CO concentration in the smoke to be followed during one puff: *cf.* WILLIAMS & BELK (22). People who are known to use this method [AYRES (9); GAISCH (13)] are satisfied with it, even if it is slightly less accurate than GC.

Finally, an automatic method for determining CO, puff by puff, after the smoke from 20 cigarettes has been collected (smoked simultaneously in line) is described by COLLINS & WILLIAMS (11).

II.1 - Nitrogen Oxides NO (B.P. = -151·7°C); NO_2 (B.P. = +21·3°C):

Routine determination of nitrous oxide N_2O [mentioned, for example, by PHILIPPE & HACKNEY (32)] are not carried out. It is not very toxic and not easily oxidisable under ambient conditions.

Nitric oxide NO, the major component of higher nitrogen oxides (NO_x = NO + NO_2) in native smoke [NORMAN & KEITH (30); BARKEMEYER & SEEHOFER (24); WILLIAMS & BELK (37)] partially oxidises fairly quickly to nitrogen peroxide NO_2 under ambient conditions [BARKEMEYER & SEEHOFER (24); SLOAN & KIEFER (34)]. Finally, in the gas phase, the amount of detectable NO_x decreases very rapidly the longer it is kept. It has been shown that certain known components of the gas phase can play a part in this disappearance of NO, as they react with it: these are: acetaldehyde, isoprene and hydrogen

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sulphide [NORMAN & VINSON (31)], and methanethiol [SLOAN & KIEFER (34)]. It would therefore be desirable to have a method which does not suffer from interferences and permits almost instantaneous analysis after one cigarette is smoked, or, better, after each puff.

The most satisfactory method for NO, from the point of view of rapidity and specificity (allowing for the negligible amounts of NO_2 in native smoke) would doubtless be the luminescence produced by the reaction $NO + O_3 \longrightarrow NO_2 + O_2$. Unfortunately, the apparatus indispensable for this method is costly (about $10,000).

Gas phase chromatography is only used for smoke by HORTON *et al.* (28), who use a 1/4 inch column packed with 28% Pennwalt 223 and 4% KOH on "Gas-Chrom R" 80-100 mesh, and an electrolytic conductivity detector. Their results agree "reasonably well" with those obtained by line spectrometry.

All the other methods described consist of:

a) either immediately determining the nitrogen oxides in the "native" gas phase

b) or contacting them immediately with a reagent having more rapid kinetics than the other smoke components in their reaction with NO_x.

The methods of type a) used in practice are:

- Non-dispersive IR spectrometry: WILLIAMS & BELK (37). A study of the possible interferences shows that, apart from ethylene, isoprene and acetaldehyde, the components of the gas phase are not very troublesome, and that errors in measurements of NO due to their presence (including that of the above 3 compounds) are no greater than about 6%.

- UV Spectrometry: The method described by URBANIC & SUTT (36) (λ = 227.25mμ: after the Cambridge filter, the smoke passes through a filter of special activated carbon, which reduces the interferences due to the other components of the gas phase) can be used for routine analysis [AYRES (23)] and permits measurements repeated 3 times in 40 minutes, puff by puff. The only two drawbacks to the method (apart from the price of the apparatus, which is in fairly general use) are: rather complicated calculations (to take into account the ratio of the volume of the measurement cell : volume of puff, and the disappearance of about 10% of the NO from the cell between 2 successive puffs) and, above all, the necessity of a suitable activated carbon as the use of some types of carbon tends to result in the disappearance of a substantial portion of the NO (*cf.* URBANIC & SUTT).

The methods of type b) actually used are:

- Selective electrode for nitrate ions: after oxidation of NO_x to NO_3^- by 1% hydrogen peroxide H_2O_2, at pH 3·5 [SLOAN & MORIE (35)]. The method is not expensive (if the laboratory already has a pH meter), but necessitates stirring for 30 minutes to oxidise NO and NO_2 to N_2O_5 (hydrolised to NO_3^-), which is slower than spectrometric methods (but other smoke samples may be taken during this time, as the smoking machine is free).

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- Colorimetric methods: used in most of the work on smoke before 1969. They have the advantage of only requiring cheap apparatus in general use, and could doubtlessly be automated fairly easily.

The only specific method published for NO is to complex it with ferrous sulphate $FeSO_4$ (coloured complex with λmax = 445nm) described by BARKEMEYER & SEBHOFER (24), who consider its relative standard error to be less than 4%.

Most authors use the SALTZMAN method (33) for determining NO_2: NO must therefore be oxidised to NO_2 assuming that, during the time necessary, the unoxidised NO does not react with other compounds of the gas phase, which is not certain (31)(34). As in the technique using a selective electrode, the gas phase is collected in a vacuum flask containing a sufficient amount of reagent. In about 2 hours the oxygen of the reintroduced air oxidises NO to NO_2 which is converted into NO_2^- (a mechanism which is not well understood and is certainly complex); NO_2^- diazotises sulfanilic acid and the diazo couples with an aromatic amine, usually N(α-naphtyl)-ethylene diamine, to give a mauve-pink compound (λ max = 545nm), stable for longer than 24 hours. The method has many drawbacks: questionable reproducibility; doubtful value (and apparently variable with the concentration of NO_x) of the coefficient to be used to pass from molar concentrations in nitrite ion NO_2^- (generally used for calibrating for reasons of convenience) to the concentration of oxide NO_2; slow kinetics for the formation of the dye. To improve the method (for mixtures other than cigarette smoke) it has been suggested: to add an arsenite AsO_2^-, which collects the OH radicals formed by the reaction $NO_2 \longrightarrow NO_2^-$ in a basic aqueous medium (and which interferes with this formation by producing NO_3^- ions) [CHOVIN & DELAUNAY (26)];

- or, instead of using nitrate as a standard, using a mixture having a known NO or NO_2 content [FISHER & BECKNELL (27); BULTEZ (25)];

- NO can be oxidised to NO_2 with ozone [FISHER & BECKNELL (27)] or oxygen [BULTEZ (25)], or again by passing the gas through a filter impregnated with bichromate $Cr_2O_7^{2-}$ [KURODA (29)], chromic oxide CrO_3, or permanganate $KMnO_4^-$;

- finally, according to FISHER & BECKNELL, by adding 1% boric acid H_3BO_3 to the reagent, it is possible to almost completely eliminate the interferences due to ammonia NH_3 (up to 25 ppm.).

Unless it is substantially improved, there will be a tendency for laboratories wishing to make a large number of measurements with good reproducibility to give up this method.

II.3 - Acrolein (B.P. = +52·5°C):

Despite LAURENE & HARBIN's (44) affirmation that there is no loss of acrolein on the Cambridge filter, it is possible that, given its boiling point, it condenses on it to a certain extent. But no method described up to now makes it possible to determine it accurately in total smoke: all measurements are made on the gas phase "for convenience".

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The following methods are used::

- Gas chromatography:

The first authors used conventional columns but, under certain conditions, it is very difficult to separate acrolein from furan [AYRES (39); TESTA & JOIGNY (46)], except for cryothermal chromatography [HORTON & GUERIN (43a)]: trapping of the gas phase is achieved on the column at -75°C; analysis time is about 1.5 hr. At present, most of the laboratories which determine acrolein directly by GC use glass capillary columns, like ARTHO & KOCH (38), in a method adapted from GROB (43). For such a method, it is naturally necessary to have a good technique for manufacturing capillary columns and to be ready to accept that analysis should take quite a long time (in the range of 30 to 60 minutes) to bring out the peaks of all the components of the gas phase (it is obviously possible to temperature programme more rapidly in order to elute the higher boiling compounds without attempting to recover them, but this procedure may rapidly degrade the column).

Another problem is the length of time smoke is kept before being injected: injection of one puff (from one cigarette or from several smoked simultaneously) appears to be more reliable than amalgamating the gas phase of a whole cigarette although, according to GROB (2), a stainless steel - teflon syringe hardly denatures the gas phase at all. When a single puff is injected the problem arises of the number of the puff, so that it is representative of the whole of the smoke from the cigarette (the analysis takes too long for each puff to be analysed successively). A reasonable solution would seem to be the use of a certain number of cigarettes smoked simultaneously, with one or two puffs displacement between them, the injection being done on a fraction of the mixture of all their gas phases. But this system poses a problem of the smoking machine, and perhaps also of the mixing chamber before the injection (see § II.6).

- Colorimetric method using 4-hexyl resorcinol according to COHEN & ALTSCHULLER (40):

The gas phase is cold trapped in the reagent solvent (ethanol) in the dark. This method has the drawback of being difficult to perfect, and of using toxic reagents (corrosive mercury chloride, trichloracetic acid); replication is not always excellent, and the amounts of acrolein added to the trapping solution are not recovered integrally [cf. DONZEL (41)].

- Gas phase chromatography of the addition compounds of acrolein:

a) The addition of labelled cysteine GAISCH (42), followed by electrophoresis and two-dimensional paper chromatography permit a determination of acrolein and of many other compounds.

b) For adding to an alkylthiol [TESTA & JOIGNY (46)], gas phase may be trapped on a water-impregnated chromosorb column, under conditions in which it appears that acrolein does not have a tendency to be destroyed, and separation of the addition products obtained (for compounds with an activated double bond, particularly acrolein) from other compounds of the gas phase is greatly facilitated compared with the starting products, owing to be relatively large increase in the mass. Conventional columns can thus be used, and chromatography lasting for about ten minutes for acrolein and in the order of 1/2 hour for all the visible products. This method has the drawback of necessitating a reaction with kinetics which are quite difficult to control, and the advantage of permitting the introduction of an internal standard, which is much more difficult with a direct injection of the gas phase.

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- It should be noted that the method of chromatography on Porapak described by MORIE & SLOAN (45) has been abandoned by its authors (for reasons of homogenicity of batches of the porous substrate) for a method using capillary columns.

II.4 - Hydrogen cyanide HCN (B.P. = 26°C)

Hydrogen cyanide is distributed in variable proportions (notably depending on the types of tobacco) between the gas phase and particulate phase (which retains 25 to 50% according to authors). However, the same methods of trapping can be used for total smoke or the gas phase (the only difference being the presence or absence of a Cambridge filter behind the cigarette) and the methods described here are valid in both cases.

Trapping is done either on a solid adsorbent: ascarite (or any asbestos containing soda) [COLLINS *et al.* (50), KUHN & BRAUN (52), REIF (55)] or silica gel behind a Cambridge filter [COLLINS *et al.* (51)] (both filters are connected for the determination of HCN, the silica gel only being reserved for the determination of volatile aldehydes), or in a scrubber containing an aqueous caustic soda solution [ARTHO & KOCH (47), AYRES (48)].

Determination is then effected by three principal types of methods:

- Colorimetric methods:

They use:

- either a bromination of the cyanide ion in an HCl medium (the excess of bromine is destroyed by arsenic oxide As_2O_3 or an arsenite AsO_3^{3-}), followed by a coloured reaction in a pyridine medium with benzidine [ARTHO & KOCH (47)], p-phenylene diamine [according to BARK & HIGSON (49), AYRES (48), MORIE (54)] or barbituric acid [KUHN & BRAUN (52)];

- or, in an automated method, chlorination of the cyanide ion with chloramine T in a basic buffer solution, followed by a coloured reaction with methylphenyl pyrazolone in the presence of pyridine [COLLINS *et al.* (50)]. Acetaldehyde and hydrogen sulphide, which might interfere, do not appear to be a hindrance at the levels at which they exist in smoke.

All these methods, and particularly the last, give acceptable results on the level of reproducibility, providing the same conditions of trapping and smoking are always respected (cigarette and ambient air moisture contents have a marked influence on HCN yield).

- Cyanide ions selective electrode [VICKROY & GAUNT (57), REIF (55)]:

This method has the advantage of simplicity and, for laboratories possessing a pH meter, only necessitates buying the electrode (cost # $400). The possible interference of sulphide ions can be eliminated by the addition of a concentration of lead nitrate higher than that of the sulphide ion [VICKROY & GAUNT (57)].

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It should be noted that, apart from the authors of the two publications mentioned, who consider the method to be satisfactory, other laboratories [TESTA (56), MORIE (54)] gave it up after several trials, as they obtained values too far removed from those obtained with colorimetry, and/or as it was insufficiently reproducible. Furthermore, this method requires fairly frequent calibrations.

- Potentiometric method [MATTINA (53)]:

This method uses the reaction of silver ions Ag^+ with cyanide ions CN^-; it necessitates a pH meter, a glass electrode and a silver electrode, but does not require calibration and, according to its author, gives rise to no interference, as the precipitation potential has a fairly specific value. It further permits hydrogen sulphide to be determined simultaneously (*cf.* § II.5). But it is probably not easy to automate.

II.5 - Sulphur compounds H_2S (B.P. = -60·1°C), COS (B.P. = -50·2°C), CS_2 (B.P. = +46·5°C), and SO_2 (B.P. = -10°C):

As these compounds may play a part in the taste and the toxicity (SO_2) of smoke it could be an advantage to have simple methods for determining them. There may be mentioned:

- For H_2S alone:

- The MATTINA method (53), already mentioned and criticised with respect to HCN;

- A method using the coloured reaction (absorption at 665nm) of H_2S (trapped in a 2·5M soda solution containing 0·5% zinc acetate) with p-amino N,N-dimethylaniline sulphate (in aqueous HCl solution) and ferric chloride [AYRES (58)]; impregnation of the Cambridge filter with citric acid is recommended to avoid any absorption of H_2S;

- A sulphide ion selective electrode method [MORIE (60)], which has the advantage of giving rapid results (about 2 minutes) on a gas phase solution (trapped in a 1 M NaOH solution in the presence of 0·01 M ascorbate as antioxidant);

- For H_2S, COS, CS_2 and SO_2:

A GC method on a conventional column packed with special silica, with flame photometry detection [HORTON & GUERIN (59)]. The authors recommend a teflon smoking circuit instead of a stainless steel one, which appears to catalyse a destruction of H_2S (probably by reaction with other components of the gas phase) attaining 50% in 4 seconds. They also note a substantial increase in the mean molecular weight of sulphur products after the gas phase has been stored for 30 seconds in a teflon loop (and *a fortiori* in stainless steel). These last remarks prove that determination is fairly difficult, even on native smoke.

Moreover, for all these compounds, there are few data in the literature on their distribution in the particulate and gas phases.

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II.6 - Determination of Various Components of the Gas Phase by High Resolution Gas Phase Chromatography (GC):

For overall comparison of the smoke from several cigarettes, it may be an advantage to obtain the values for a certain number of their components in less than one hour. In the present state of the techniques, GLC on a conventional packed column does not permit sufficiently satisfactory separations of the products, even in the gas phase, although it is a very simple mixture compared with total smoke. On the other hand, according to GROB (43), the use of capillary columns permits a semi-quantitative description of the gas phase (values of concentrations of about ten or twenty products in about 1 hour and with a maximum error of about 20%). Criticism of determinations in the gas phase are naturally justified here by the large number of products having a relatively high boiling point (110° for toluene) among the compounds determined, which poses the problem of their distribution between the gas and particulate phases. If it suffices to consider such a "determination" as an empirical description of an arbitrary, but reproducible, fraction of smoke, interesting indications can be obtained for studies of filters, of new manufacturing techniques, for instance. Technically speaking, this method necessitates sufficient experience working with glass capillary columns.

A noteworthy qualitative application of the method has recently been described by ARTHO & KOCH (61).

"Quantitative" adaptations of GROB's technique are used in a certain number of laboratories: REIF (65); DONZEL & TESTA (62), who now determine 11 products and who now use a smoking machine with 4 synchronous pistons to produce a "mean" gas phase; GAISCH (63) determined 14 products; HJERN *et al.* (64), who use pressure programming which further improves separation as well as temperature programming from -70°C.

III - CONCLUSION

Although it is always desirable to have recourse to several methods for a determination - one checking the other in case of doubt - , it is often convenient, especially when a laboratory starts a new determination, to have an idea of the advantages and drawbacks of each method, and to be able to judge it as a function of local requirements (frequency of determinations, accuracy desired, apparatus available). Although this review may be rather long, an effort has been made to make it as complete and objective as possible. It has shown that, although satisfactory methods exist for quick, accurate and reliable determination of CO and HCN, progress still needs to be made, and conditions specified, for NO_x, H_2S and acrolein. We have not mentioned light unsaturated hydrocarbons, among which ethylene is an ideal component of the gas phase (B.P. = -102.4°C): as far as we know only one recent case of determination (by IR) has been effected, presented by VILCINS at the 27th TCRC (October 1973).

The critical, documentary aim of this study has not been fully attained owing to the fact that only a few laboratories (actually 5) agreed to reply to a detailed questionnaire on the methods they used

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for determining compounds in the gas phase considered to be interesting, and those they have tried and given up. The remainder of the information comes from publications - which are always behindhand compared with authors' subsequent experiments. In spite of this, we hope that this review will, as it stands, be of use to certain colleagues and will make a modest contribution to increasing exchanges within the Smoke Group.

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Appendix II

DETERMINATION OF WATER IN TOBACCO WITH THE DESTILLATION METHOD USING CYCLOHEXANE; REPORT ON A COLLABORATIVE STUDY

by

G. Lipp and H. Böttcher

Between November 1973 and February 1974 a collaborative study was made between 17 laboratories, all members of Tabakkolloquium, to examine the destillation method for the determination of water in tobacco after replacing benzene by cyclohexane. This study included three different types of cut reg: virginy, blend and stems different in moisture. One laboratory prepared all the samples using air tight tins for delivery. Three tins of each of the three tobaccos were investigated in duplicate runs by all the laboratories, i.e. in total six single values per lab.

The main results of the statistical evaluation are given in the tables 1 to 4 showing significant lower mean values for three of the 17 laboratories obviously due to individual reasons. The results of the other 14 labs are in good correspondence within a range of 0,33 % moisture. In the meantime further improvement of the method could be achieved, therefore we are convinced that this method is suitable for a CORESTA Standard Method.

Remarks:

(1) a complete report of this collaborative study will be published in the near future in "Beiträge zur Tabakforschung";

(2) an exact description of the procedure will be available within several weeks.

J. Lipp

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table 1

mean values of tins
(averages of two determinations each)
(all values in % water)

tobacco		virginy			blend			stems		
tin		1	2	3	1	2	3	1	2	3
lab	1	14,01	13,99	14,21	12,13	12,16	12,07	15,07	15,13	15,06
	2	13,74	13,50	13,59	12,27	12,20	12,13	15,86	15,69	15,63
	3	13,83	13,72	13,80	12,12	11,97	12,22	15,55	15,80	15,88
	4	14,00	14,03	14,13	12,41	12,48	12,18	15,94	16,02	16,12
	5	14,20	14,07	14,13	12,53	12,41	12,30	15,97	15,95	15,98
	6	14,35	13,85	14,00	12,50	12,50	12,65	15,85	15,95	16,00
	7	14,18	14,08	14,10	12,50	12,58	12,43	15,88	15,98	16,03
	8	14,25	14,21	14,10	12,35	12,35	12,24	16,12	16,20	16,00
	9	14,19	14,03	14,27	12,52	12,39	12,28	15,90	16,10	16,19
	10	14,04	14,21	14,16	12,48	12,33	12,43	16,05	16,11	16,29
	11	14,10	14,20	14,25	12,60	12,70	12,20	16,00	16,00	16,20
	12	14,15	14,10	14,27	12,37	12,59	12,42	16,16	16,06	16,36
	13	14,26	14,35	14,19	12,61	12,55	12,65	16,24	16,23	16,29
	14	14,43	14,45	14,32	12,56	12,39	12,46	16,18	16,34	16,26
	15	14,49	14,59	14,63	12,51	12,44	12,42	16,06	16,30	16,33
	16	14,52	14,35	14,33	12,75	12,70	12,70	16,25	16,30	16,30
	17	14,55	14,30	14,50	12,80	12,70	12,55	16,10	16,30	16,50

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table 2

mean values of tobaccos and laboratories

(all values in % water)

tobacco	virginy	blend	stems	laboratory mean
lab 1	14,07	12,12	15,09	13,76
2	13,61	12,20	15,73	13,84
3	13,78	12,10	15,74	13,88
4	14,05	12,36	16,03	14,15
5	14,13	12,41	15,97	14,17
6	14,07	12,55	15,93	14,18
7	14,12	12,50	15,96	14,20
8	14,19	12,31	16,11	14,20
9	14,16	12,40	16,06	14,21
10	14,13	12,41	16,15	14,23
11	14,18	12,50	16,06	14,25
12	14,17	12,46	16,26	14,30
13	14,27	12,60	16,25	14,37
14	14,47	12,47	16,26	14,40
15	14,57	12,46	16,23	14,42
16	14,40	12,72	16,28	14,47
17	14,45	12,68	16,30	14,48
tobacco mean	14,17	12,43	16,02	14,21

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Source: https://www.industrydocuments.ucsf.edu/docs/pkxk0000

table 3

standard deviations of tobaccos and laboratories
(all values in % water)

tobacco	virginy	blend	stems	laboratory mean
lab 1	,121	,045	,037	,078
2	,121	,067	,116	,104
3	,056	,125	,172	,127
4	,068	,156	,090	,111
5	,065	,115	,015	,076
6	,256	,086	,076	,162
7	,052	,075	,076	,068
8	,076	,063	,100	,081
9	,122	,120	,148	,130
10	,088	,073	,127	,099
11	,076	,064	,115	,172
12	,087	,115	,264	,174
13	,080	,050	,032	,057
14	,047	,085	,080	,072
15	,072	,047	,147	,098
16	,104	,028	,028	,064
17	,132	,125	,200	,156
tobacco mean	,106	,110	,125	

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Source: https://www.industrydocuments.ucsf.edu/docs/pkxk0000

table 4

statistical evaluation of significant differences between laboratory mean values (95 %)

(all values in % water)

lab	$\bar{x}$	17	16	15	14	13	12	11	10	9	8	7	6	5	4	3	2	1
17	14,48																	
16	14,47																	
15	14,42																	
14	14,40																	
13	14,37																	
12	14,30																	
11	14,25																	
10	14,23																	
9	14,21																	
8	14,20																	
7	14,20																	
6	14,18	+																
5	14,17	+	+															
4	14,15	+	+															
3	13,88	+	+	+	+	+	+	+	+	+	+	+	+	+				
2	13,84	+	+	+	+	+	+	+	+	+	+	+	+	+	+			
1	13,76	+	+	+	+	+	+	+	+	+	+	+	+	+	+			

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Source: https://www.industrydocuments.ucsf.edu/docs/pkxk0000

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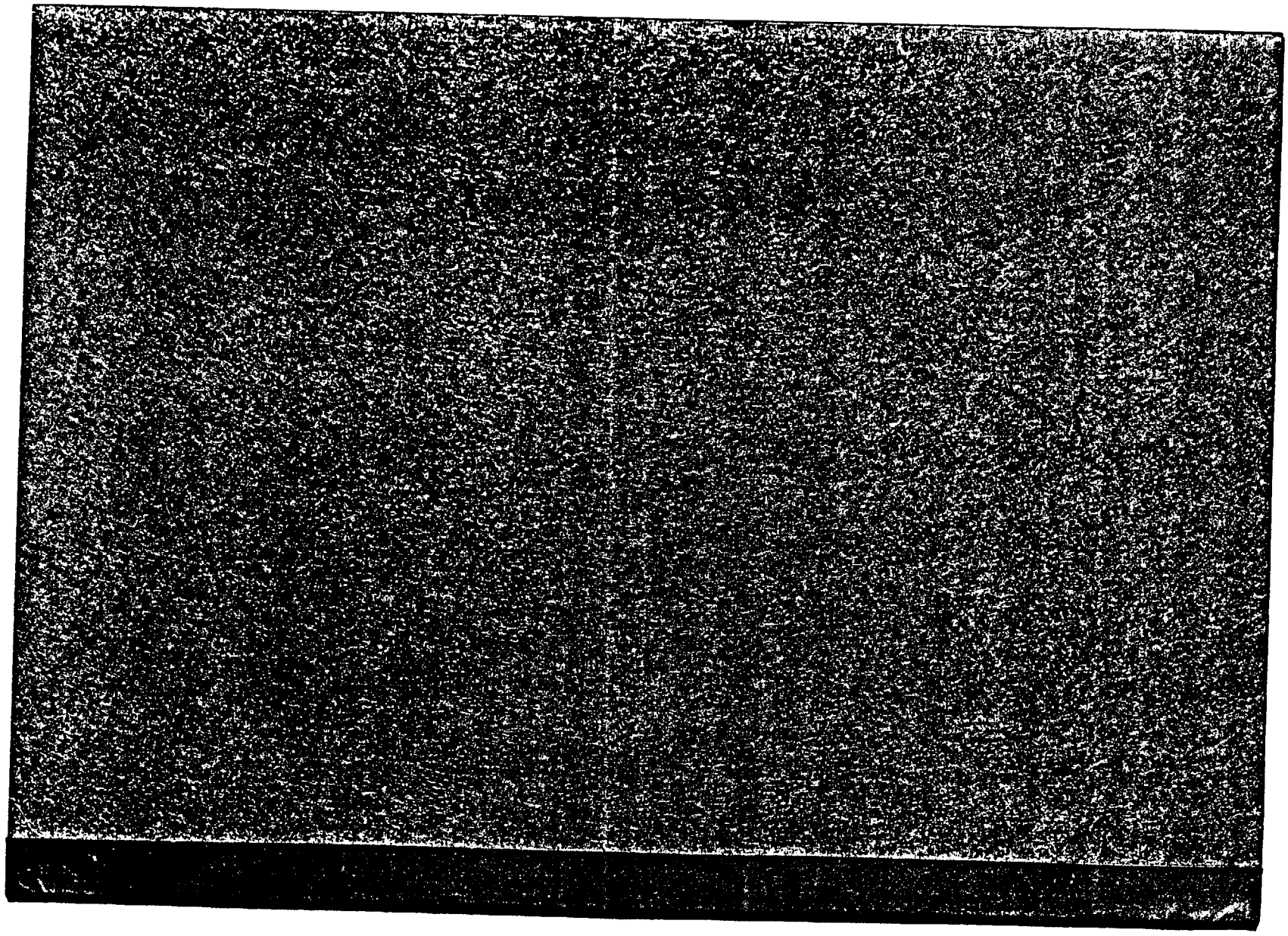

Source: https://www.industrydocuments.ucsf.edu/docs/pkxk0000

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Source: https://www.industrydocuments.ucsf.edu/docs/pkxk0000